Lowering of photocatalytic activity of TiO_2 particles during oxidative decomposition of benzene in aerated liquid

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ABSTRACT

TiO_2-photocatalyzed reaction was conducted in pure benzene in which TiO_2 particles were suspended. Benzene was oxidatively decomposed into CO_2, as most of organic compounds. However, the rate of CO_2 evolution from benzene was gradually lowered as the reaction continued, which was in contrast to the cases of other non-aromatic organic compounds, such as alcohols or acids. To clarify the reason for the decrease in activity of TiO_2, intermediates produced in the solution phase and on the surface of TiO_2 were analyzed using chemical and physical techniques. Several kinds of intermediates were identified: phenolic compounds, aldehydes, carboxylic acids, and polymeric substance. Of these products, catechol and polymeric substance, which were observed only on the surface of TiO_2 powder, were found to be responsible for the lowering of the photocatalytic activity of TiO_2.

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1. Introduction

Photocatalysis is a promising way of utilizing solar energy and maintaining the environment. Among various available photo-catalysts, TiO_2 has been attracting much attention due to its good stability and high activity. Many kinds of harmful organic substances can be mineralized into CO_2 and water by TiO_2-photocatalyzed reaction [1–4]. It would be very meaningful to construct a system for decomposing benzene or volatile aromatic compounds using photocatalysts because these compounds are highly toxic and carcinogenic and their concentrations in the environment are strictly limited. However, there are some reports on difficulty of decomposition of aromatic compounds using TiO_2 photocatalysts [2–5]. More precisely, aromatic compounds are decomposed more slowly than other organic compounds. In addition, during the photocatalytic reactions, the reaction rate is further lowered and TiO_2 powders are colored. Adsorption of intermediate products is thought to be the reason for the deactivation and coloration of TiO_2 particles, both of which are problematic for the practical application of TiO_2 photocatalysts.

Surface charge transfer (CT) complexes formed between TiO_2 and phenolic compounds have been proposed as the cause of coloration of TiO_2. Alberici and Jardim [2] suggested that the species adsorbed on TiO_2 were responsible for the deactivation of TiO_2 photocatalysts during photocatalytic reactions of aromatic compounds. However, they did not determine the products. Einaga et al. [3] analyzed the deactivated TiO_2 particles by means of IR spectroscopy and found "carbon deposits" on the surfaces of the particles. d'Hennezel et al. [5] extracted the species adsorbed on the TiO_2 surface with organic solvents and detected phenol, hydroquinone, and benzoquinone as intermediate products from benzene. However, Wu et al. [6] reported that species with phenoxy groups were formed on the TiO_2 surface and suggested that the products extracted from TiO_2 with organic solvents may not reveal the real products formed on the surface. We found that the products extracted with organic solvents are not the main cause of the deactivation and coloration of TiO_2 because, as described later, the TiO_2 powder after extraction of these intermediates products is colored and shows lowered photocatalytic activity. This means that there are considerable amounts of intermediate products that are not extracted from the TiO_2 surface by using conventional organic solvents.

We consider the identification of these products, which cannot be extracted with organic solvents, are important for the clarification of the deactivation of TiO_2 photocatalysts when used in the presence of aromatic compounds. In this study, therefore, we analyzed the products formed in liquid and on the surface of TiO_2 in photooxidation of benzene. Based on obtained results, we discuss the cause of deactivation and coloration of the TiO_2 photocatalyst when used for the oxidation of benzene.

2. Experiments

TiO$_2$ powders ST-01, ST-21 and ST-41 obtained from Ishihara Sangyo, Ltd., NS-51 from Toho Titanium Co. Ltd., TIO-3 from Ishihara Sangyo Co. Ltd., MT-150A from Teika Co. Ltd. and P-25 Degussa from Nippon Aerosil Co. Ltd. were used as photocatalysts. Contents of rutile phase and relative specific surface area of these TiO$_2$ powders are as follows: ST-01: 0%, 192.5 m^2/g; ST-21: 0%, 56.1 m^2/g; ST-41: 0%, 8.2 m^2/g; MT-150A: 100%, 110 m^2/g; TIO-3: 100%, 48.1 m^2/g; NS-51: 98.5%, 6.5 m^2/g and P-25: 26%, 49.2 m^2/g.

Benzene, phenol, catechol, hydroquinone, resorcinol, oxalic acid, maleic anhydride, malonic acid, acetic acid, sodium fluoride, and hydrogen chloride obtained from Wako Pure Chemical as guaranteed reagents were used without further purification.

All photocatalytic reactions were carried out at room temperature in 40 ml Pyrex glass tubes (1.5 cm in diameter) containing 100 mg of the TiO$_2$ powder and 5 ml of benzene. The gas phase of the tubes was filled with air. A 500 W high-pressure mercury lamp (Wacom BMO-500DY) was used as a light source, and the light beam was passed through a 34-filter (Kenko Co.) to eliminate deep UV components ($\lambda < 340$ nm) and through stainless-steel meshes to lower the light intensity to about 0.6 mW/cm^2. The reaction suspension was magnetically stirred during the reaction.

CO$_2$ evolution in the gas phase was monitored with a Shimadzu GC-8A gas chromatograph. Liquid phase was analyzed by an HPLC system equipped with a Hitachi L-7400 UV detector and 4.6 mm $\times$ 150 mm columns (GL-Sciences, Intersil ODS-3). The HPLC mobile phase was a mixture of acetonitrile and water (volume ratio of 1:1) with a flow rate of 0.5 ml/min. The products were identified from the results of HPLC by comparing retention times and UV–vis adsorption spectra of the products with those of corresponding authentic compounds.

The TiO$_2$ powder samples that had been separated from the reaction solution and dried in ambient air at 80 °C for 3 h were analyzed for identification of adsorbed products. FTIR spectra of solid samples were recorded on a Nicolet Nesus 370S IR spectrometer with resolution of 2 cm^{-1}. UV diffuse reflectance spectra were obtained with a Shimadzu UV-2450 spectrophotometer equipped with an integrating sphere. For thermogravimetry (TG) and differential thermal analyses (DTA) of the samples, 20 mg of the powder sample was put in an opened pan in air and analyzed with a Bruker Thermal Analyzer (AXS TG-DTA 2000SA). Temperature was raised at a rate of 15 °C/min. The products adsorbed on the TiO$_2$ powder were also analyzed after they were extracted from the TiO$_2$ surface. For the extraction, 25 mg of dried powder after photocatalytic reaction was ultra-sonicated in 2 ml of aqueous solution containing NaF (0.08 M) and HCl (0.72 M) for 2 h at 40 °C, and then the liquid was analyzed by HPLC.

The adsorption isotherm of catechol on the TiO$_2$ powder was obtained as follows. TiO$_2$ powder (100 mg) was added to each of the catechol solutions in toluene (10 ml) with different concentrations. The suspensions were ultra-sonicated for 45 min at 40 °C to reach the equilibrium, and the concentration of catechol in the solution was determined by HPLC.

3. Results and discussion

3.1. CO$_2$ evolution from benzene and products in liquid phase

When organic compounds are decomposed by TiO$_2$-photocatalyzed reaction, CO$_2$ evolves. Time courses of CO$_2$ evolution from benzene samples containing different TiO$_2$ powders are shown in Fig. 1. High CO$_2$ evolution rates were obtained by using ST-01, ST-21, and P-25 powders. They have large specific areas and are chiefly made of an anatase phase. The high activity of P-25 powder may partly be attributed to the synergism between rutile
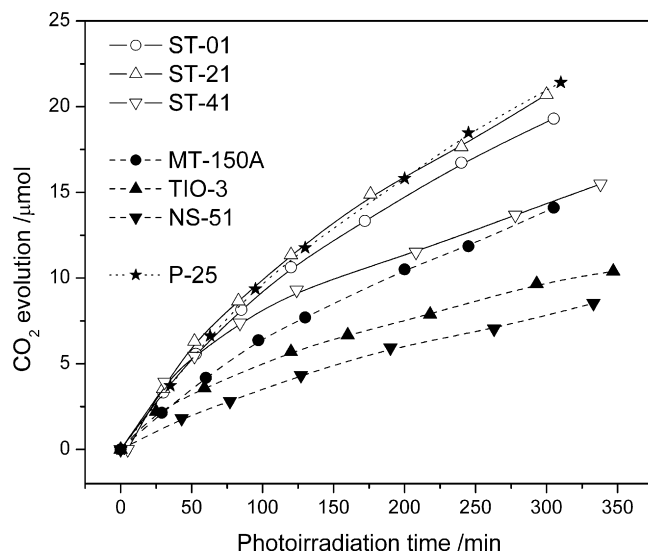

Fig. 1. Time courses of CO$_2$ evolution from pure benzene with several kinds of TiO$_2$ powders as photocatalysts.

and anatase phases [7]. In all cases, the CO$_2$ evolution rate decreased with irradiation time, and the tendency was obvious for the powders with small specific surface areas (ST-41, TIO-3, and NS-51). This tendency suggests that intermediate products produced from benzene have negative effects on the photocatalytic activity of TiO$_2$ surface. With the production of these intermediates, the TiO$_2$ particles gradually changed from white to pale yellow and, then, brown, as discussed later.

The lowering of the CO$_2$ evolution rate with irradiation time was observed when benzene or some aromatic compounds were used as the samples, whereas the rate was constant for most of other organic materials as long as the components of the liquid do not change remarkably. To demonstrate the difference, the time courses of CO$_2$ evolution from pure benzene and pure acetic acid are compared in Fig. 2.

The lowering of CO$_2$ evolution rate from benzene with time suggests that some of the intermediate products from benzene have harmful effects on photocatalytic activity of TiO$_2$ particles. Therefore, we tried to determine products dissolved in liquid and also those adsorbed on TiO$_2$ surface. In the following experiments, we mostly used ST-01 powder as photocatalyst.
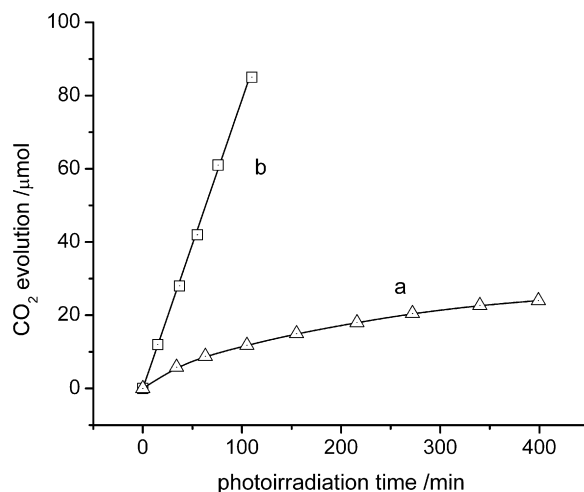

Fig. 2. CO$_2$ evolution by photocatalytic reaction of (a) pure benzene and (b) pure acetic acid using TiO$_2$ powder (ST-01) as a photocatalyst.
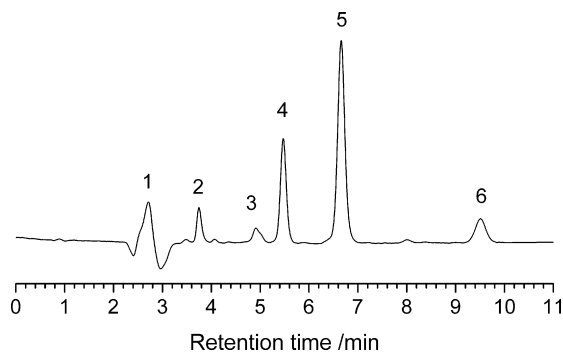
Fig. 3. A typical HPLC chromatogram of benzene after photoirradiation using TiO_2 powder (ST-01) as a photocatalyst.

In order to determine intermediate products from benzene, we periodically sampled small aliquots of the reaction liquid (benzene) during the photocatalytic reaction and analyzed them by HPLC. Typically, 6 peaks (Peaks 1–6) were observed in the HPLC chromatograms, as shown in Fig. 3. The valley observed after Peak 1 is an artifact, which appeared even when pure benzene was injected into the HPLC system. By comparing the retention times and UV–vis spectra of the components with those of the authentic samples, four of these peaks were assigned to p-hydroquinone (Peak 2), muconaldehyde (Peak 3), p-benzoquinone (Peak 4), and phenol (Peak 5). These compounds were reported as products from benzene by the TiO_2-photocatalytic reaction [3,5,8]. Although Peak 1 was not identified, it is probably due to an acid or a mixture of acids produced from benzene; possible acids such as oxalic acid and formic acid, which can be produced from benzene, showed retention times almost at the same position. Peaks 1–5 grew for 30 min during photoirradiation and then started to decrease, whereas Peak 6, which appeared at the longest retention time, continued to grow. This last peak is probably due to dimeric species, i.e., hydroxybiphenyls and/or aromatic ethers. Production of these dimeric compounds from phenol in the TiO_2-photocatalyzed reaction was reported by Peiró et al. [9] and Ng et al. [10].

3.2. Analysis of products adsorbed on TiO_2

The deceleration of CO_2 evolution is probably related to the intermediate products which are not only dissolved in the liquid phase but also adsorbed on the TiO_2 surface. The adsorption of these intermediates was evident from coloration of the TiO_2
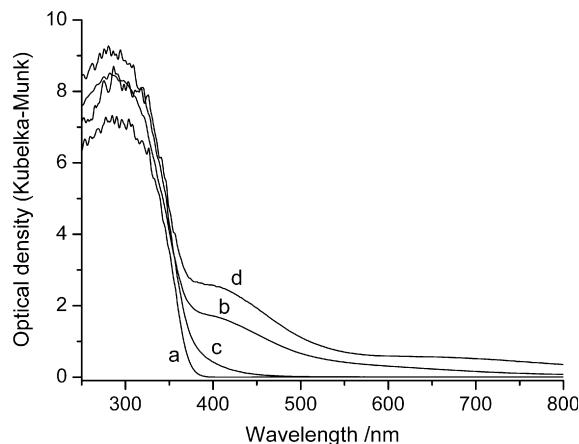

Fig. 4. Typical diffuse reflectance spectra of TiO_2 powder (ST-01) (a) before and (b) after photoirradiation for 35 min in benzene. Spectra (c) and (d) are of the TiO_2 powder on which phenol and catechol were adsorbed, respectively.
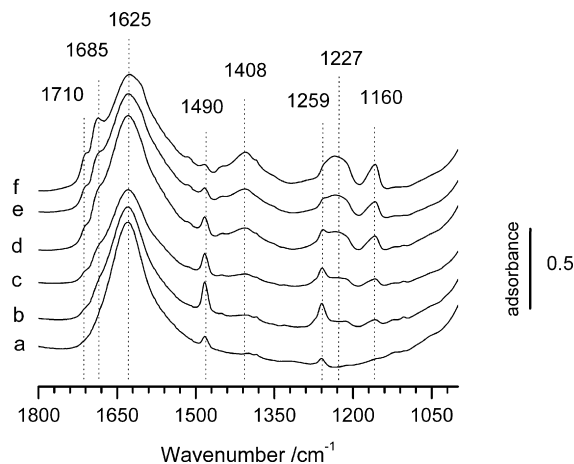

Fig. 5. FTIR spectra of TiO_2 (ST-01) powder after photoirradiation in benzene for (a) 10 min, (b) 120 min, (c) 240 min, (d) 360 min, (e) 480 min, and (f) 600 min.

particles during photoirradiation. For analysis of the intermediates, the TiO_2 powder after photoirradiation was separated from benzene and dried in air. Diffuse reflectance spectra of the photoirradiated TiO_2 powder and the same powder before photoirradiation are shown by curves (a) and (b) in Fig. 4. The results indicate that the intermediate products exist not only in the liquid but also on the surface of TiO_2.

Since the species adsorbed on TiO_2 powder are considered to have a stronger influence on the photocatalytic activity than those dissolved in solution, identification of the adsorbed species on TiO_2 is important. We therefore measured FTIR spectra for KBr discs containing the TiO_2 powder after photoirradiation. With increase in the irradiation time in benzene, two peaks at 1490 and 1259 cm^{-1}, which are typical peaks of C–C stretching vibration of benzene rings and C–O stretching vibration, increased and then started to decrease after photoirradiation for 240 min, as seen in Fig. 5. These peaks are due to phenolic compounds adsorbed on TiO_2 [6,11]. Three peaks at 1710 and 1685 and 1408 cm^{-1} became clear after 240 min. The peaks at 1710 and 1685 cm^{-1} are characteristic of asymmetric and symmetric stretching vibration of C=O double bonds, respectively [12]. The shift of these peaks to lower frequencies than normal positions suggests that these carbonyl groups are connected with a conjugated system or adsorbed on the surface of TiO_2. The peak at 1408 cm^{-1} should be due to C–O–H bending. These results suggest that some acids are gradually formed on the surface of the TiO_2 powder. Two peaks observed at 1227 and 1160 cm^{-1}, which grew with irradiation time, are probably due to polymeric compounds formed from phenolic compounds on photoirradiated TiO_2 particles [10,13].

In order to identify the species adsorbed on TiO_2 after photoirradiation, we extracted them from the TiO_2 surface by immersing the TiO_2 powder after photoirradiation using organic solvents. We detected phenol, hydroquinone, and benzoquinone, as reported by d'Hennezel et al. [5]. However, they cannot be the main cause of the deactivation and coloration of TiO_2 particles because the TiO_2 particles after extraction were strongly colored and showed a low level of photocatalytic activity.

The species strongly adsorbed on TiO_2 particles must be identified to clarify the reasons for the deactivation and coloration of TiO_2. It is known that F^- is strongly adsorbed on TiO_2 and that TiO_2 gradually dissolves in HF solution [14,15]. Hence, the colored TiO_2 after photocatalytic reaction was immersed in an aqueous solution containing NaF (0.08 M) and HCl (0.72 M) for 30 min. Dissolution of TiO_2 was negligible during the treatment. The products extracted into the solution were analyzed by HPLC. There were two main products in the HPLC chromatogram, which were
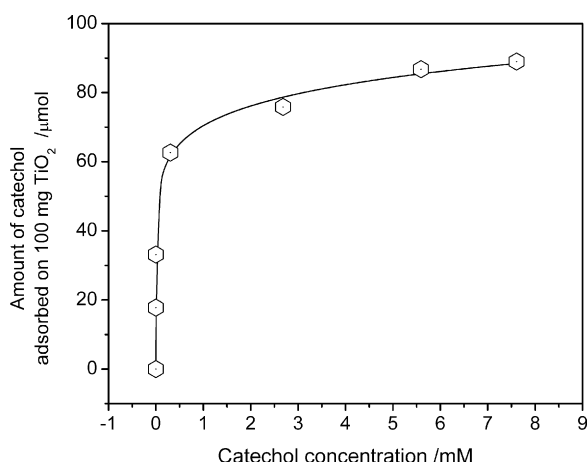
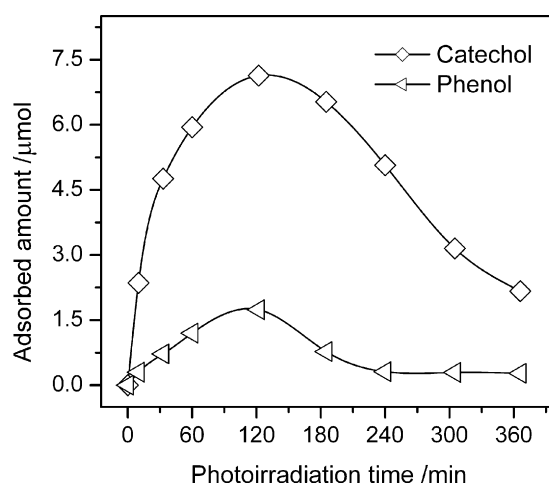Fig. 6. Adsorption isotherm of catechol on TiO$_2$ (ST-01).

Fig. 7. Time course of production of catechol and phenol on TiO$_2$ (ST-01) powder. The products were extracted from dried samples (100 mg) of TiO$_2$ photoirradiated in benzene.

assigned to phenol and catechol, respectively. Catechol has not been detected as a product from benzene by TiO$_2$-photocatalyzed reaction [5,6], probably due to the strong adsorption of catechol on TiO$_2$. Besides these species, several unidentified products were detected in the extraction solution. Judging from the retention times and UV–vis spectra, we believe that they were carboxylic fragments produced by ring-opening reactions of benzene. Phenol was observed in large amounts both on TiO$_2$ and in benzene. In contrast, catechol was not found in solution phase, but detected in a larger amount as an adsorbed product on the surface of TiO$_2$. This is attributed to strong affinity of catechol to TiO$_2$ surface, which was proved by the adsorption isotherm of catechol, as shown in Fig. 6.

Fig. 7 shows amounts of phenol and catechol extracted from the ST-01 powder surface with increase in irradiation time. The amounts increased with increase in irradiation time in the early stage of the reaction, reached maximum values at about 120 min, and then started to decrease. This tendency is consistent with the tendency of the FTIR peaks at 1490 and 1259 cm^{-1}, which we attributed to phenolic compounds, observed for TiO$_2$ powders after photoirradiation, as shown in Fig. 5.

It is notable that the coloration of TiO$_2$ particles remained even after extraction with the solution containing NaF and HCl, although the coloration after the treatment was much less. The coloration after the treatment was not reduced or eliminated by extraction with organic solvents. These results suggest that an insoluble substance such as a polymer remains on the TiO$_2$ surface after extraction with the solution containing NaF and HCl.

Appearance of photoabsorption bands in the visible region after adsorption of catechol and phenol on TiO$_2$ powder, as shown in Fig. 4, suggests that they are chemisorbed on the surface of TiO$_2$ through the hydroxyl groups. The coloration of the TiO$_2$ particles is attributed to CT transition between the chemisorbed species and TiO$_2$ [11,16–18]. Liu et al. [16] and Rodriguez et al. [19] reported strong chemisorption of catechol on TiO$_2$. The strong adsorption of catechol on TiO$_2$ was supported by the adsorption isotherm as shown in Fig. 6. The two hydroxyl groups of catechol are thought to be responsible for its strong interactions with TiO$_2$. Presence of catechol on TiO$_2$ is important when the photocatalytic activity of TiO$_2$ is considered because it lowers the photocatalytic activity of TiO$_2$ [20]. Dimitrijevic et al. [18] suggested the possibility of driving photocatalytic reaction under visible light by utilization of CT excitation. However, the contribution of CT excitation to the overall photocatalytic reaction is generally small [21]. We also confirmed that the rate of CO$_2$ evolution under visible light from catechol-adsorbed TiO$_2$ powder, which was suspended in benzene, was very slow.

The products adsorbed on the photoirradiated TiO$_2$ powder were also analyzed by DTA in air. In the early stage of the photocatalytic reaction, two exothermic peaks were observed at 240 and 340 °C, the former being the dominant in the initial state, as shown in Fig. 8a. With increase in reaction time, the peak at 240 °C increased and then started to decrease. The peak observed at 340 °C continued to increase with increase in reaction time and became the dominant peak. The DTA curves for the TiO$_2$ powders on which phenol, catechol, oxalic acid, and malonic acid were adsorbed are shown in Fig. 8b. From a comparison of these curves, the peak observed at 240 °C in Fig. 8a is attributed to phenolic compounds. The peak observed at 340 °C in Fig. 8a is probably due to polymeric substances because its change with time is similar to the change in the FTIR peaks at 1227 and 1160 cm^{-1}, which we attributed to polymeric substances produced from phenolic compounds.

From the TG measurements we found that amounts of the products increased rapidly in the initial stage of the photocatalytic
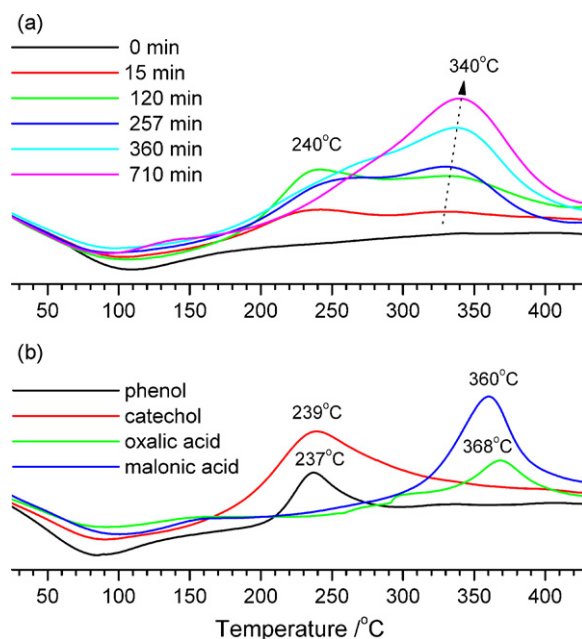

Fig. 8. (a) DTA curves for TiO$_2$ (ST-01) powder before and after photocatalytic reaction of benzene for different periods and (b) DTA curves for TiO$_2$ (ST-01) powder on which phenol, catechol, oxalic acid or malonic acid were adsorbed from aqueous solutions.
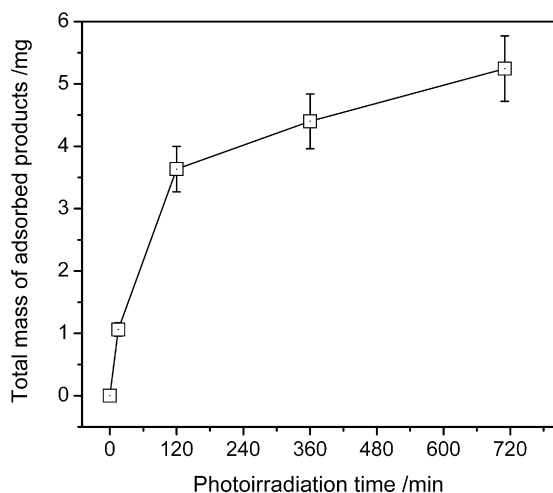
Fig. 9. Total mass of products adsorbed on 100 mg of TiO$_2$ (ST-01) powder. The mass was determined by TG measured over a temperature region from room temperature to 450 °C; the mass change for the control sample was subtracted.

reaction and then increased at a much slower rate after photoirradiation for about 100 min, as shown in Fig. 9. The rapid increase is probably due to the adsorption of phenolic compounds and acids, whereas the slow increase after 100 min is probably due to the accumulation of polymeric substances.

From all of the above-described results, we conclude that there are three main products that are adsorbed on the photoirradiated TiO$_2$: phenolic compounds, carboxylic acids, and polymeric substances.

3.3. Effects of intermediate products on photocatalytic activity of TiO$_2$

To evaluate the effects of some compounds on the photocatalytic activity of TiO$_2$ for oxidation of benzene, we added phenol, hydroquinone, catechol, resorcinol or maleic anhydride to benzene and monitored the amount of CO$_2$ evolved during photoirradiation. The results in Fig. 10 show that the rate of CO$_2$ evolution was lowered by addition of phenol, catechol, hydroquinone, or resorcinol. In contrast, carboxylic acids such as maleic anhydride were easily oxidized on TiO$_2$ under photoirradiation, causing no lowering in the rate of CO$_2$ evolution. The largest deceleration effect was observed when catechol was added, and the effect decreased in the order of catechol, phenol, hydroquinone, and resorcinol. When 60 μmol of catechol was added to 5 ml of benzene containing 100 mg of TiO$_2$, the evolution of CO$_2$ was totally suppressed. It has been reported that the strong surface complexation of catechol with TiO$_2$ allows it to cover the TiO$_2$ surface and that the adsorbed catechol resists oxidation by holes [20]. The suppression of CO$_2$ evolution by the addition of 60 μmol of catechol is attributed to this effect. Evolution of CO$_2$ was observed when the amount of catechol added was 5 μmol probably because there remained a surface area that was not covered by catechol. In this case, the initial rate of CO$_2$ evolution was about 5 μmol/h. When photocatalytic reaction was carried out in authentic benzene, a similar reaction rate was observed after photoirradiation for about 60–75 min, as seen in Fig. 10. With this photoirradiation time, the amount of catechol adsorbed on TiO$_2$ (100 mg) was also about 5 μmol, as shown in Fig. 7. These results strongly suggest that deactivation of TiO$_2$ particles was due to the adsorption of catechol which was produced from benzene. However, the profile of the production of these species, as shown in Fig. 7, is not consistent with the deceleration of the photocatalytic reaction for a longer time period; the reaction rate decreased even when the amounts of catechol and phenol started
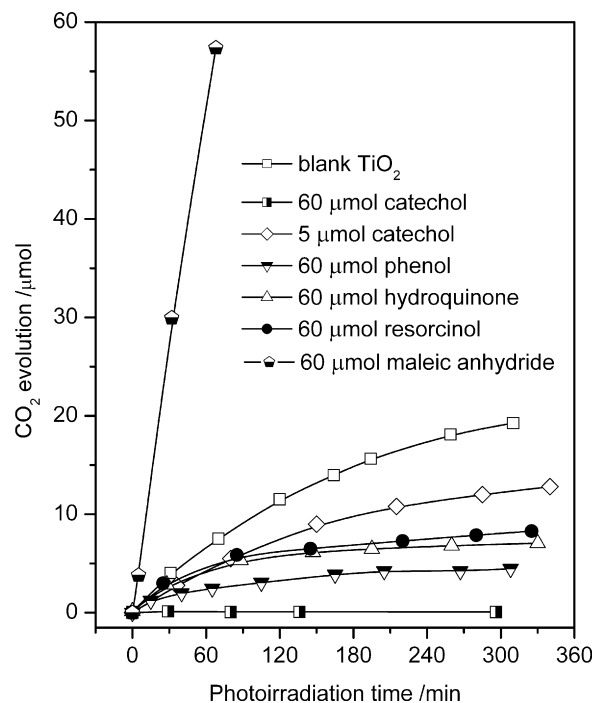

Fig. 10. CO$_2$ evolution by TiO$_2$ (ST-01) photocatalyzed reactions of pure benzene and benzene containing phenol, hydroquinone, catechol, resorcinol, or maleic anhydride. The amounts of the compounds added to 5 ml of benzene are shown in the graph.

to decrease. This suggests that the polymer substance is also harmful to the photocatalytic reaction. This may be due to the recombination of electron–hole pairs on the polymer or a strong photoabsorption by the polymer material. The results obtained also suggest that the processes on TiO$_2$ change with time as the photocatalytic reaction proceeds.

The deceleration of CO$_2$ evolution by phenol, catechol, and polymer compounds means that they have a negative feedback on oxidation of benzene. A complete analysis of the kinetics is difficult because the profiles of the changes in the amounts of phenol, catechol, and polymer compounds are complicated. However, the time courses of changes in the CO$_2$ evolution rate (Figs. 1 and 2) and in amounts of adsorbed products (Figs. 7 and 9) suggest that the apparent rate constant for the decomposition of polymer compounds is lower than those for the decomposition of catechol and phenol. In other words, in the initial stage of photoirradiation, when the amount of polymer compounds is not large, adsorption of catechol on the TiO$_2$ particles is the chief reason for the deceleration of CO$_2$ evolution. As the amount of polymer compounds increases, because of its negative feedback on the oxidation of benzene on photoirradiated TiO$_2$ particles and its slow decomposition rate, the amount of adsorbed catechol starts to decrease. Instead, the amount of polymer compounds continues to increase. Therefore, the polymer compounds will be responsible for the deactivation of the TiO$_2$ particles.

Finally, the relation between the results described above and gas-phase reactions should be discussed. In most practical applications of TiO$_2$ photocatalysts, they are used in a gaseous atmosphere containing very low concentrations of organic compounds under irradiation with a much lower photon density. In the case of photocatalytic reaction carried out in air containing benzene molecules, TiO$_2$ powder gradually became colored after a long time. This suggests that compounds determined in authentic benzene in the present study are also produced in gas-phase reactions. However, the production of polymer compounds may not be significant in gas-phase system because the reaction rate is

usually very slow due to the low concentration of benzene. Investigation is now being carried out to identify the colorant formed in the gas-phase reaction of benzene.

3.4. Conclusions

We analyzed intermediate products from benzene in TiO$_2$-photoctalyzed reactions. The products formed in solution were phenol, p-hydroquinone, p-benzoquinone, muconaldehyde, some acids, and dimeric or trimeric substances of hydroxybenzenes. On the surface of TiO$_2$, besides these compounds, catechol and a polymeric substance were formed. These substances adsorbed on TiO$_2$ surface were found to have a great effect on the photocatalytic activity.

Acknowledgements

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